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Y-1654

Engineering
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AEC RESEARCH AND DEVELOPMENT REPORT

HIGH-DENSITY TUNGSTEN SPHERES

J. H. Nash
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UNION CARBIDE CORPORATION
NUCLEAR DIVISION
OAK RIDGE Y-12 PLANT

operated for the **ATOMIC ENERGY COMMISSION** *under* **U. S. GOVERNMENT** **Contract W-7405 eng 26**

**UNION
CARBIDE**

OAK RIDGE Y-12 PLANT
P. O. Box Y
OAK RIDGE, TENNESSEE 37830

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U.S. Department of Commerce, Springfield, Virginia 22151
Price: Printed Copy ~~\$2.00~~, Microfiche ~~\$0.65~~

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Date Issued: November 18, 1968

Document Y-1654

Engineering and Equipment
TID-4500

UNION CARBIDE CORPORATION
Nuclear Division

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Contract W-7405-eng-26
With the US Atomic Energy Commission

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Document Y-1654Engineering and Equipment
TID-4500

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ABSTRACT

Two, four-inch-diameter, unalloyed tungsten spheres were machined and lapped to a sphericity of better than five microinches and a background surface finish of less than three microinches, peak to valley. The material used was a special, uniform-density, gas-pressure-bonded tungsten made specially for the job.

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SUMMARY

*typical
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Three, four-inch-diameter spheres of a very uniform, high-density, unalloyed, gas-pressure-bonded tungsten were fabricated for use in an experiment designed to provide a more precise value of Newton's gravitational constant. Two of the spheres were lapped to a sphericity of better than five microinches and a background surface finish of three microinches, peak to valley. The average density of the spheres, which varied less than 300 $\mu\text{gms/cc}$ between themselves, was 19.074 gms/cc . The mass-center dislocation from the geometrical center was less than 300 microinches.]

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INTRODUCTION

The University of Virginia needed a pair of uniform, high-density tungsten spheres for an experimental determination of Newton's gravitational constant.^(a) In addition to the need for uniform density, a very precise geometry and surface finish were required. Specifications called for a nominal 4.000-inch diameter with a sphericity of ten microinches or better. Assuming no voids or inclusions, 90 percent of the surface finish was to be one microinch or less, peak to valley. Although the absolute diameter was not critical, it was desirable to have the two spheres as closely matched as possible without going to extreme measures or excessive cost.

(a) This work was done under Purchase Order 12392.

THE FABRICATION PROCESS

MATERIAL CHOICE

The requirements for these parts were that they have a high and very uniform density, be dimensionally stable, and be reasonable in cost. Unalloyed tungsten was chosen by a process of elimination. All alloys were unacceptable because a variation in density between the separated volume cells was predictable. Uranium was unacceptable because of its oxidation and subsequent dimensional change; other high-density elements, such as rhenium, were too expensive for the application.

BILLET FABRICATION

Having settled on tungsten as the best material, the method of fabrication needed to be resolved. Two processes were open: (1) machining from a high-density sintered billet, or (2) machining from a forged billet. A survey of the nation's tungsten forgers showed that they were not optimistic concerning the second method. One vendor proposed to undertake the task on a best-effort basis for approximately \$50,000 if Y-12 would supply the starting material "from Allied Chemical Corporation's gas-pressure-bonded tungsten". This statement was significant in that Y-12 proposed to supply the sintered machining billet from this material if the density uniformity was acceptable. Consequently, test parts, one inch in diameter and three inches long, were made from granules sized between 350 and 420 microns. These parts were deemed acceptable and the large parts were ordered.

At this point, Allied Chemical Corporation, for internal reasons, decided to quit the field of gas-pressure bonding tungsten and to give the process to the Government. Negotiations to make this transfer lasted over 15 months and included an agreement to make a limited number of gas-pressure-bonding runs. These runs were primarily to demonstrate the process, but one run was to be used to make the machining billet. The process engineers felt the greatest chance of success in getting density uniformity lay in taking the three required parts from one billet. This decision necessitated making a sintered billet that was 4 3/4 inches in diameter, 14 inches long, and weighed 157 pounds. This billet was, by far, the largest tungsten part ever made by gas-pressure bonding.

To make the billet, high-purity tungsten granules were sieved through a 40-mesh screen and onto a 45-mesh screen. The granules were then loaded into a multiwall molybdenum-titanium-molybdenum cylindrical pressure-transfer canister through a small hole. The loaded canister was then evacuated and the small hole closed by electron-beam welding. Consolidation was accomplished by heating the canister in a gas autoclave, shown schematically in Figure 1, to 2,900° F at a pressure of 13,000 psi and holding at these conditions for eight hours. Also included in the run

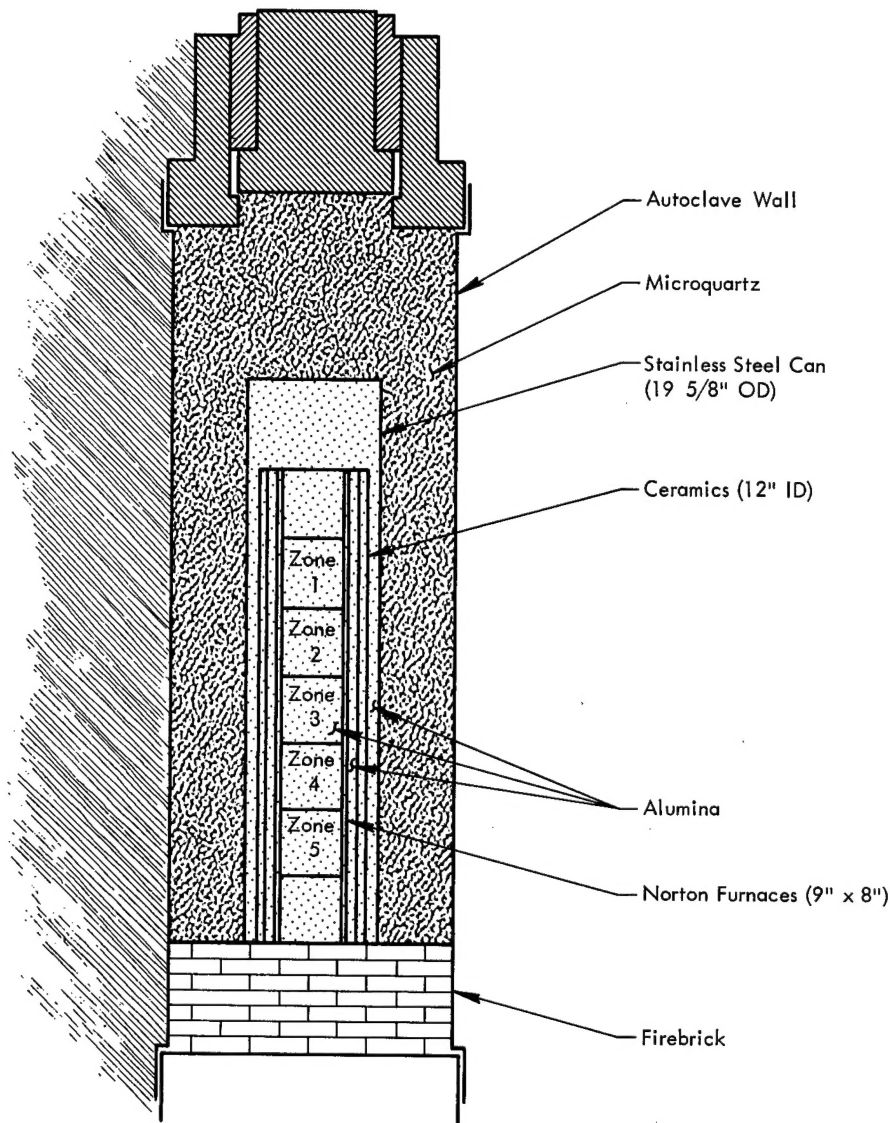


Figure 1. GAS AUTOCLAVE.

were four test cylinders. The location of these parts is shown schematically in Figure 2. Following a cool-down period, the canister was removed from the billet by acid leaching. Figure 3 shows the billet cut into three sections with one section having been rough machined. Note the wrinkles in the outside surface resulting from a dimensional change during densification. Figure 4 is a close-up view of the surface and clearly shows the outer granular surface. Figure 5 shows the microstructure of one test part (T-542) containing the same tungsten granules as the large billet and consolidated at the same time.

The open-end type of furnace, shown in Figure 2, leads to an uneven hot zone; however, the part was loaded in a manner expected to take full advantage of the entire length of the hot zone. Unfortunately, the bottom apparently projected into

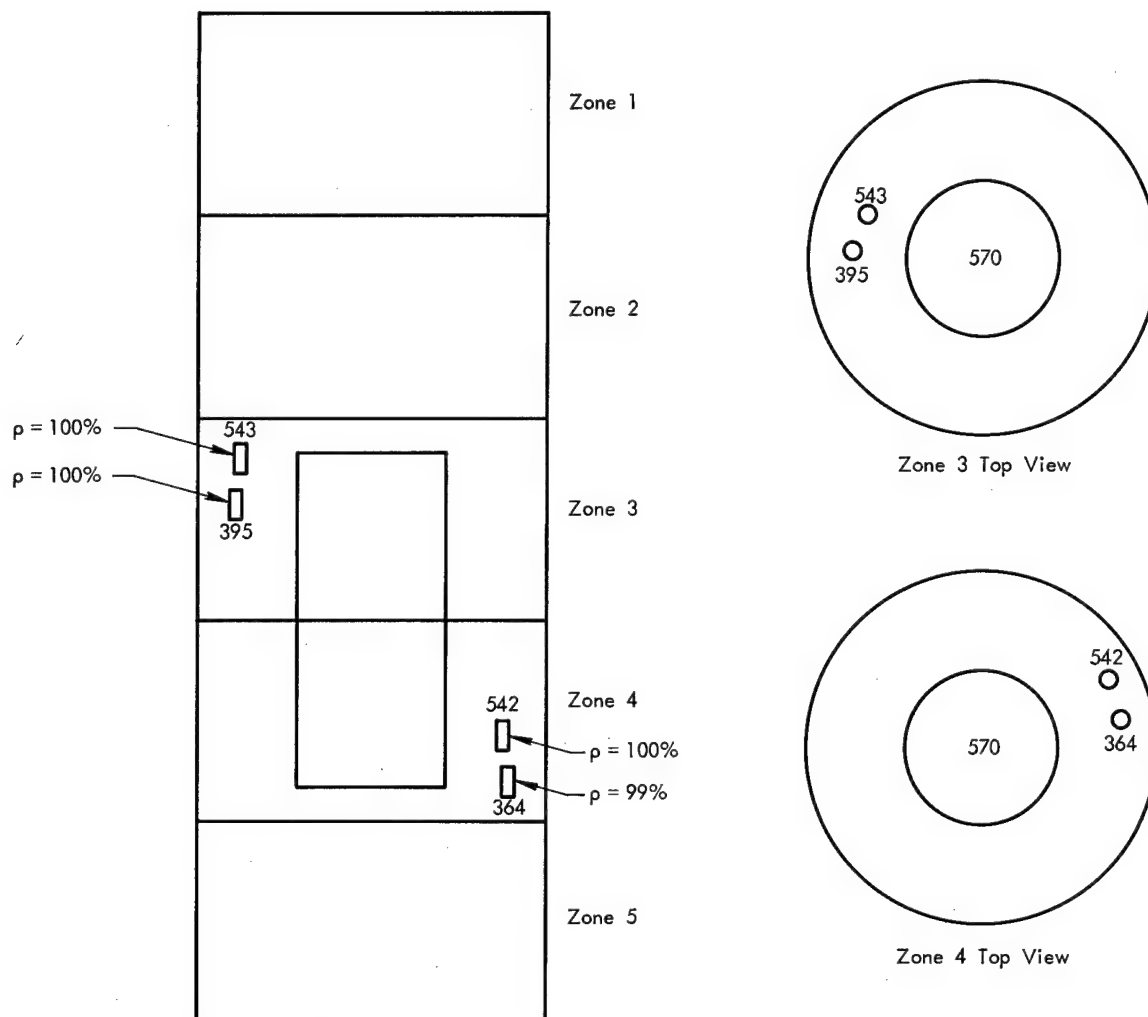


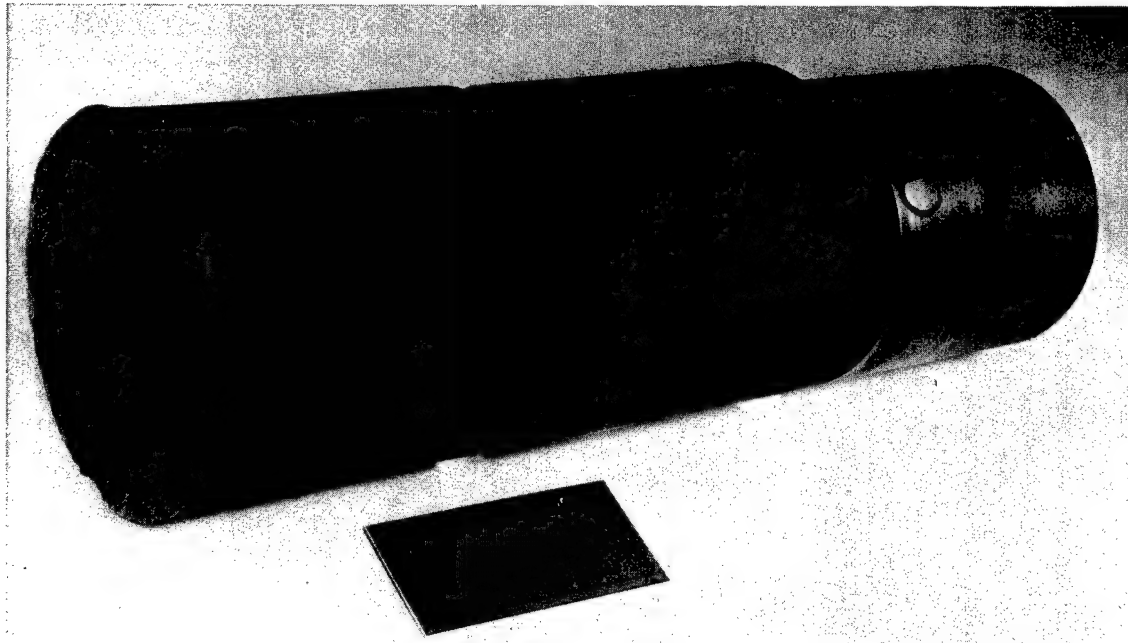
Figure 2. FURNACE-LOADING ARRANGEMENT. (p = % of the TD)

a colder zone, producing a lower-density portion within the billet. The densities of the test pieces, also shown in Figure 2, confirm this supposition since the bottom test piece had the lowest density.

SPHERE FABRICATION

After completing the metallurgical studies, the tungsten cylinders were machined into spheres on a standard radius mill. Approximately 1/16 inch of excess stock on the diameter was left for finishing on a precision hemisphere turning machine which had been designed and fabricated for the Y-12 Plant to study single-point machining techniques. The machine can be seen in Figure 6.

Investigations conducted on the turning of tungsten using diamond knives⁽¹⁾ had indicated that it might be possible to turn the spheres to either the finish or very



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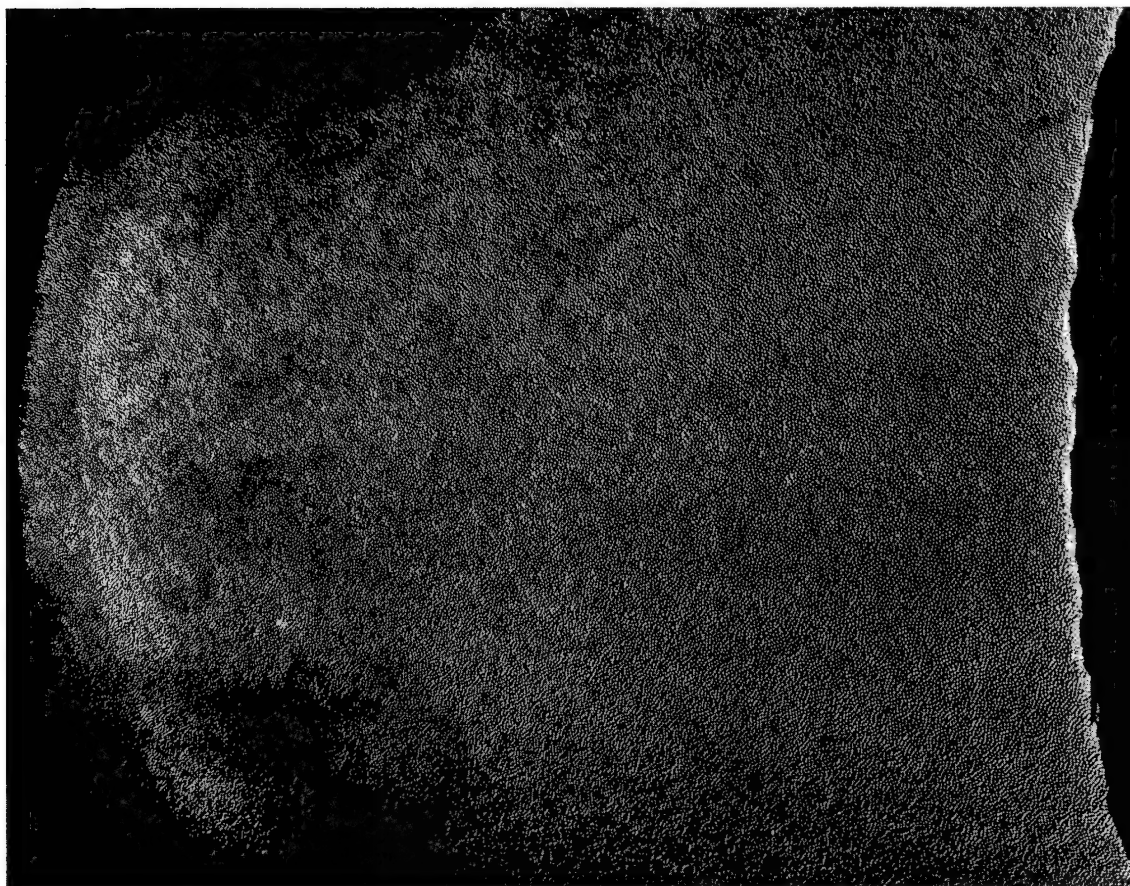
Figure 3. DECANNED TUNGSTEN, GAS-PRESSURE-BONDED BILLET. (Partially Machined)

close to the finish geometry required, thus reducing or eliminating the necessity of lapping. However, the diamond-knife machining efforts on this machine were unsuccessful and the best results were poor. Several attempts were made using a range of speeds from 100 to approximately 1,000 rpm both dry and with several different cutting fluids including trichloroethylene, EDM fluid (used to machine aluminum), and sulfochlorinated oils. In each case the diamond knife fractured after just a few revolutions. [The spheres finally had to be machined^w with carbide tools. The best results were obtained by using Grade K-68^(a) carbide insert tools, which had an 18-mil tip radius, at a speed of approximately 1,000 rpm using trichloroethylene as a cutting fluid. Several other grades of carbide tools were tried, including Grades 883 and 999^(b) with both 21-mil-radius tips and knife edges, but none were too successful.]

Final results of the machining operation produced a sphere which was round to within approximately three mils. This amount of out of roundness was due, primarily, to the excess material that was left at the pole. The two factors which contributed to the poor polar condition were: a low surface speed and tool breakdown. [Because of the poor sphericity obtained in machining, approximately 15 mils of excess stock on the diameter were left for lapping the spheres to the required geometry.]

^(a) Kennametal Inc, Latrobe, Pennsylvania.

^(b) Carboloy, Division of General Electric, Detroit, Michigan.



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Figure 4. SURFACE TUNGSTEN, GAS-PRESSURE-BONDED BILLET. (Showing Undeformed Surface of Outer Granules)

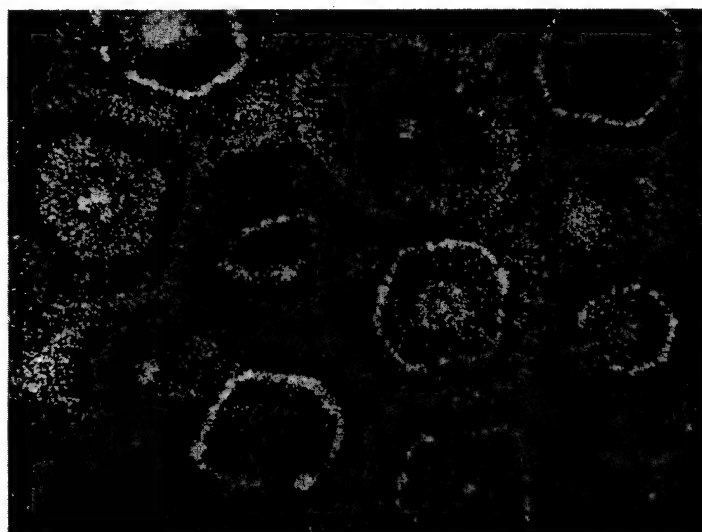
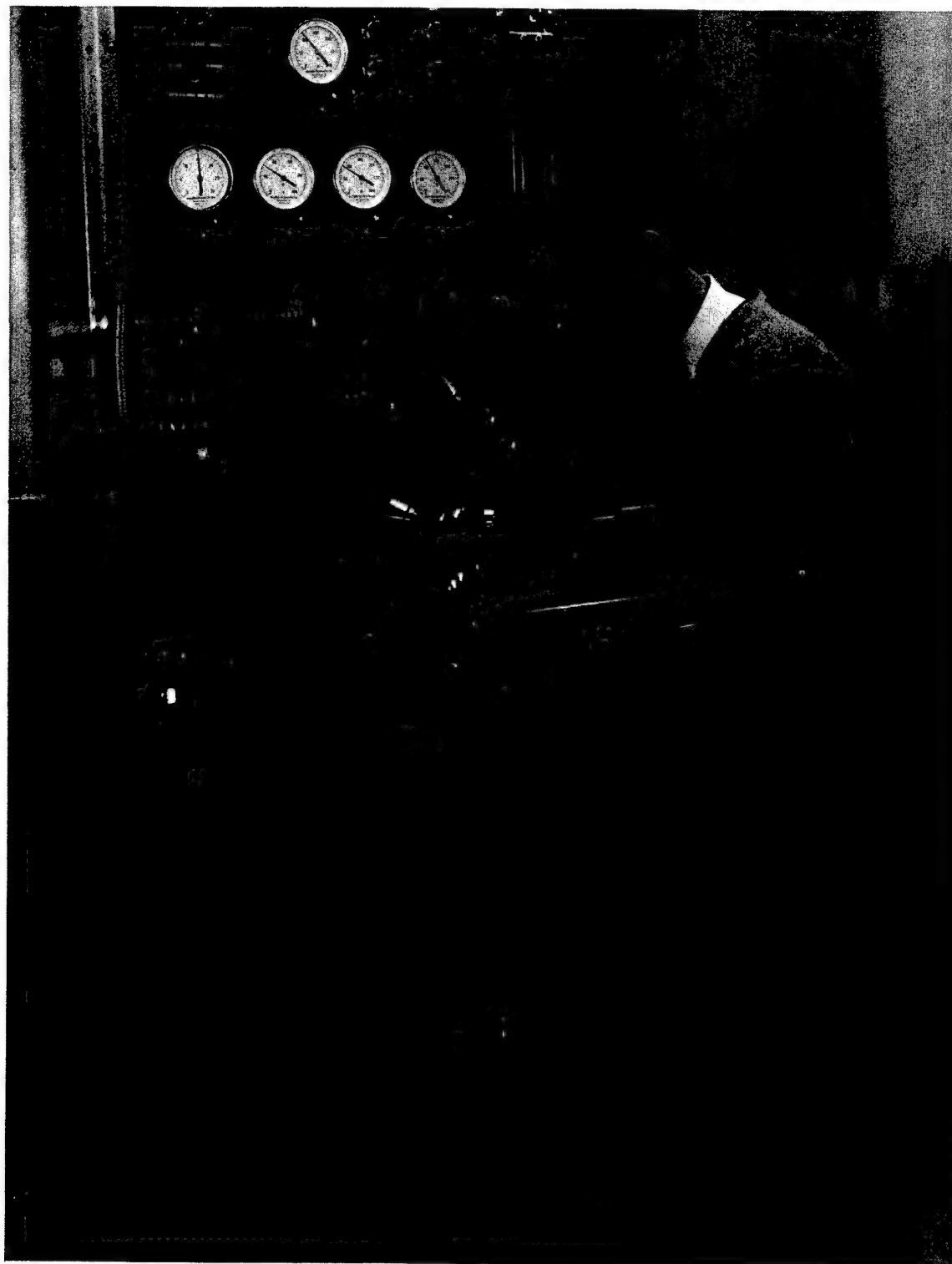


Figure 5. MICROSTRUCTURE OF GAS-PRESSURE-BONDED TUNGSTEN.



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Figure 6. HEMISPHERE TURNING MACHINE.

Finishing of the spheres was done by three-point lapping. At first, the laps were set in a vertical plane due to the weight of the spheres; however, this position was not successful as the proper motion could not be obtained. The laps were then set in a horizontal plane and a jack-screw arrangement with a Teflon pad was used to support the ball weight. This method proved to be satisfactory.

[Initial lapping of the spheres was done with 17-micron aluminum oxide and John Crane lapping oil using cast-iron laps. The spheres were brought to a sphericity of 25 microinches with this compound.] Density measurements were made at this point using the mercury-displacement technique which is done in a manner similar to that described for water. The measurements indicated densities of 19.01, 19.05, and 19.07 gms/cc for the three spheres. Since the variation was less than 0.4 percent, approximately the accuracy of the measurements, it was felt that the uniform-density requirements could be met.

[Final sphericity was obtained by using three-micron aluminum oxide compound and John Crane lapping oil with cast-iron laps. The final surface finish was obtained by first lapping with three-micron aluminum oxide and John Crane oil on glass Teflon laps and then with six, three, and one-micron diamond compound, successively, using Sunnen honing oil as a lubricant on microcloth-lined laps. Roundness measurements were made at various intervals during the final lapping stages for surface finish to insure that sphericity was maintained. Final roundness measurements indicated a sphericity of better than five microinches for two of the spheres, a sphericity 30 to 40 microinches for the third, and a background surface finish of three to four microinches, peak to valley, for all three spheres. Further attempts to round the third sphere were unsuccessful.] The reason for this failure is not certain, but a microscopic inspection of the surface of the spheres indicated a greater variation of material porosity around the surface for this sphere, compared to the other two spheres. This sphere also exhibited the lowest density, indicating that it was machined from the bottom billet which projected into the colder zone of the furnace. The finished lapped spheres are shown in Figure 7. The results of these measurements are reported in Table 1.

INSPECTION

After the roundness measurements, the final diameters of the three spheres were measured on the Du Pont research machine which is equipped with an opposed white-light interferometer measuring system. The system is referenced to a gage-block buildup. Temperature sensors were attached to the gage blocks and to the part, and the temperature in the room was lowered until the part temperature was approximately 68° F. The spheres were initially measured on a Moore measuring machine and this approximate diameter was used for the gage-block buildup. The sphere and gage-block buildup were placed on the machine with the interferometers, and the whole system was allowed to normalize.



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Figure 7. FINISHED TUNGSTEN SPHERES.

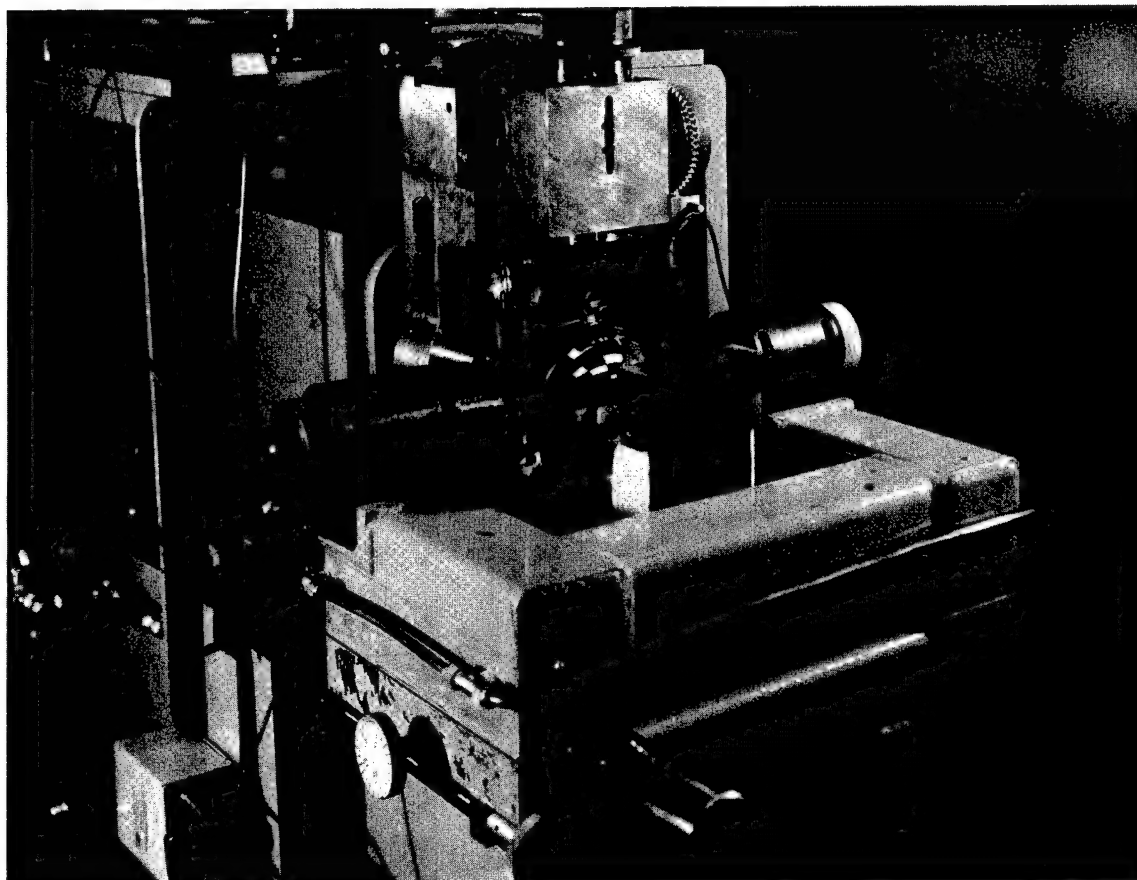
Table 1
PHYSICAL CHARACTERISTICS OF THE FINISHED TUNGSTEN SPHERES

Sphere Number	Size (inches)	Roundness Deviation (TIR) (microinches)			Balance Period (sec)	Mass-Center Dislocation from the Geometrical Center (microinches)	Surface Finish(1) (μ in)	Weight (kgs \pm 0.00007)	Average Density (gms/cc)
		1	2	3					
0038	4.001997	5	3	3	30	181.5	3	10.489980	19.073914
0397	4.002011	3	3	5	24	298.0	3 - 4	10.490250	19.074866
0037	4.000936	20	30	40	15	726.2	3	10.445982	19.009009

(1) Peak to valley.

After the temperature of the sphere and gage blocks had leveled off, the measurements were started. Each sphere was measured on three mutually perpendicular diameters. Each diameter was measured from six to ten times, monitoring the temperature each time, with a time interval of from fifteen minutes to one hour between measurements. When changing to another diameter, the part was again allowed to

normalize. From each group of readings an average diameter was found, and from the three average diameters an average diameter for the sphere was determined. The equipment arrangement is shown in Figure 8; the final measurements are listed in Table 1.



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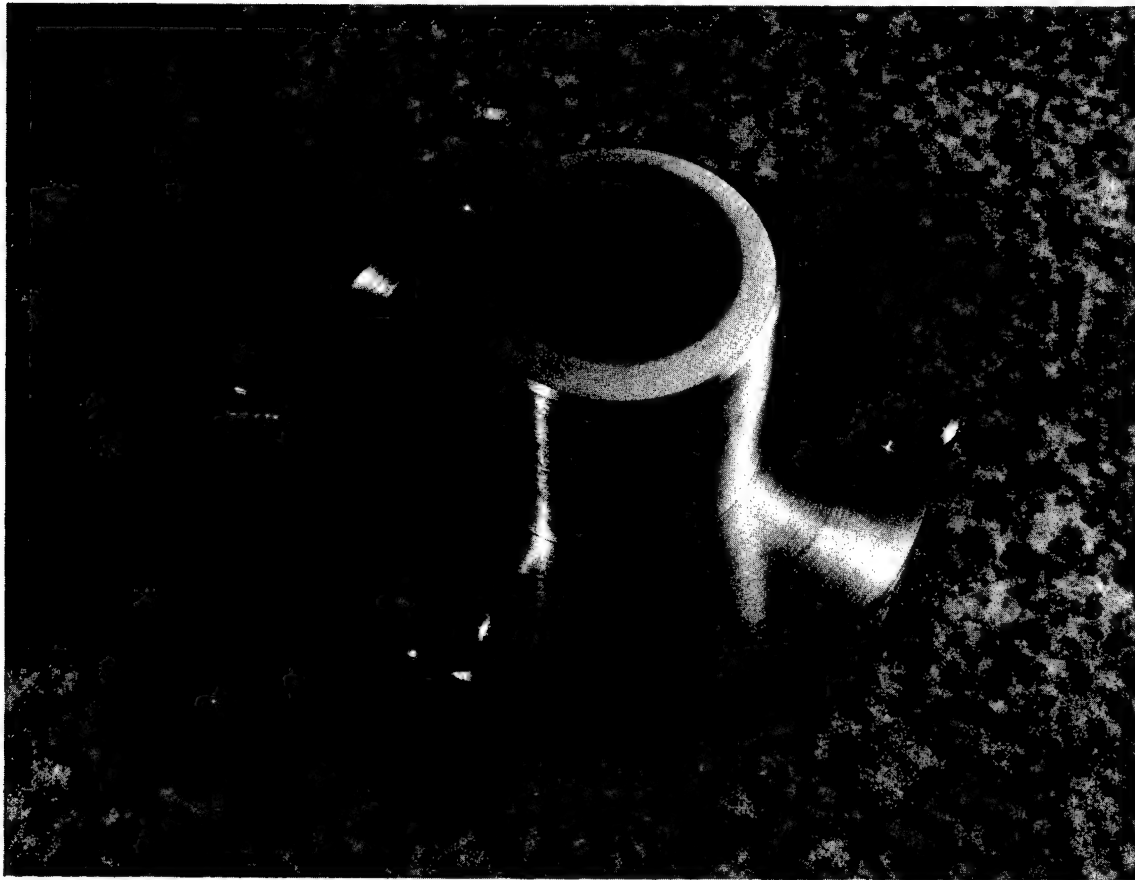
Figure 8. WHITE-LIGHT INTERFEROMETER MEASURE SYSTEM.

In taking the measurements, each interferometer was zeroed in, setting the black fringe in the center of the bullseye, on either end of the gage-block buildup. The interferometers were then moved to the sphere and, moving both as a unit, one interferometer was zeroed in on the sphere, again so that the black fringe was in the center of the bullseye. Then, through the opposite interferometer, the displacement of the black fringe was counted in fringes, estimating to one-tenth of a fringe. This method gave the difference in size between the sphere and the gage-block buildup. To determine the value of the fringe spacing, a krypton-86 orange interference filter was used when estimating the fringe fraction. For this light, one fringe was equivalent to 11.0 microinches. Temperature corrections were applied to both

the part and the gage blocks to bring them to 68° F. The fringe difference was then added in to give the final reading.

In order to establish the relationship between the mass center and the geometrical center, the balance period for the spheres was measured. The balancing device used, which was designed and fabricated for these measurements, consisted of a noncaptive, spherical-segment, externally pressurized air bearing. Porous graphite was used for the bearing pad which provided the necessary bearing compensation with maximum stability and uniform air flow. The balancing device is shown in Figure 9. Neglecting friction and viscous damping, the approximate relationship which exists between the mass center and the geometrical center was determined from the following differential equation of motion for the sphere oscillating on the bearing:(2)

$$I \frac{d^2\theta}{dt^2} = -Wh\theta,$$



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Figure 9. SPHERICAL AIR-BEARING BALANCE.

where:

I represents the mass moment of inertia,

θ the angular displacement of the gravitational force acting through the mass center and the line connecting the two centers,

W the weight of the sphere, and

h the distance between the centers.

The solution of this equation yields the following expression for determining the displacement of the mass and geometrical centers, in inches, after substituting for the moment of inertia of the sphere:

$$h = \frac{8\pi^2 r^2}{5gt^2},$$

where:

r represents the radius of the sphere, in inches,

t the balance period, in seconds, and

g the gravitational acceleration, in inches per second per second.

The sphere mounted on the balancing device for measuring the balance period is shown in Figure 10. Both the measured balance periods and calculated displacements for the three spheres are tabulated in Table 1.

The three spheres were weighed on a thirty-kilogram, two-pan analytical balance. The apparent mass of each sphere was determined from a series of intercomparisons with each other and with the primary mass standards certified by the National Bureau of Standards. Comparisons were made by the method of single transposition. The true mass of each sphere was determined from the apparent mass by calculating the effects of air buoyancy. Density was then determined from the calculated volume based on the average measured diameter and the measured true mass of the spheres. This information is also tabulated in Table 1.

Critical observation of the surface of the spheres during cleaning after completion of these measurements revealed several small nicks and scratches. Information concerning the scratches as well as the results of the measurements was sent to the principals at the University of Virginia. Two members of the University made a trip to Y-12 to view and discuss the spheres. The result of the visit was to accept the spheres in their present condition. The scratches were not considered to be critical in view of the relatively large mass-to-geometrical center displacement



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Figure 10. MEASUREMENT OF THE TUNGSTEN SPHERE OSCILLATION PERIOD.

which existed and the uncertainty of the density uniformity. Since this value was primarily a function of the material composition and structure and not geometrical accuracy, attempts to improve the condition would be doubtful and difficult. Accordingly, it was agreed that the spheres would be satisfactory for an initial experimental run. At this time also, surface finish, which was slightly poorer than desired, was judged satisfactory. Originally, the third sphere, which had the poorest geometrical accuracy, was scheduled to be machined into test specimens for examination. However, because of the many experimental uncertainties which existed, one of which was the accuracy requirements of the spheres to be used for the experiment, the third sphere will instead be used in an experimental run and the results of this run compared to results obtained using the two accurate spheres. An attempt will then be made to establish the influence of the physical characteristics of the spheres on the accuracy of the experiment.

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ACKNOWLEDGEMENTS

The authors wish to express their appreciation to all those who contributed to this project. In particular, they wish to acknowledge the efforts of the following Y-12 personnel: V. F. Chapman, Fabrication; R. R. Baldwin, Technical Services; W. F. Jones, Development; and J. O. Hylton, Maintenance.